1,005,338

PATENT SPECIFICATION

DRAWINGS ATTACHED

1,005,338

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Int. Cl.:—C 03 b, c

COMPLETE SPECIFICATION

Semicrystalline Glass-Ceramic Body and Method of Making It

We, CORNING GLASS WORKS, a corporation organized under the laws of the State of New York, United States of America, of Corning, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to glass-ceramic bodies which are dense, strong, resistant to thermal shock, and have such a high softening point that they are eminently suitable for many high temperature applications, such as furnace walls, rocket nose cones and heat exchangers.

Within the past few years, exceptional products of the glass industry have been discovered. These products can be obtained by controlled crystallisation of a glass body, thereby yielding what has generally been termed a semi-crystalline body. This discovery has opened an entirely new field of ceramics which has been called glass-ceramics. The process usually comprises a special heat treatment of a glass shape to induce the formation of very small internal crystals. In some cases, a chemical compound may be added to the glass batch to act as a nucleator for crystal growth. With a careful heating schedule, a dense, strong, ceramic-like material can be produced directly from the glass. Because it is first a glass, the new material can be formed into almost any shape by blowing, pressing, spinning or drawing. Some types can even be cast like metals. Finally, because the crystallisation is carefully controlled within a glass shape, a substantially homogeneous body of fine crystals which is substantially free of voids and is non-porous can be produced. This is in direct contra-distinction to the typical fused cast ceramic body which usually contains many voids and is coarse grained.

Recent applications for dense, uniform, high temperature ceramic materials that can be formed into various intricate shapes have severely taxed the conventional ceramic techniques and have created a rich potential for glass-ceramic materials and methods.

It has been learned that substantially all glasses having a high alumina content tend to devitrify when held for an appreciable time at a temperature slightly below their liquidus temperatures, but the rate of the devitrification depends in large measures upon the glass composition, for any given constant percentage of alumina. Binary aluminate compositions, e.g., calcium aluminate and aluminum phosphate, can be quenched to yield glasses only with considerable difficulty. Binary aluminosilicate glasses containing more than 30 weight percent of alumina are very unstable, i.e. tend to devitrify readily when cooled from the melt. However, because the refractoriness of compounds containing substantial amounts of alumina, such as mullite, is so high, their presence would be extremely desirable.

We have found that at least 35 percent by weight of alumina is necessary in an alumina-silica glass batch to ensure a substantial crystallisation of mullite and/or other high-alumina crystal phases. Therefore, to be of practical value as a refractory material, the glass should contain at least 35 weight percent of alumina. Accordingly, this invention concerns the crystallisation of refractory compounds from glasses, particularly where a high alumina compound forms the predominant crystalline phase, and wherein no nucleating agent need be utilised.

The present invention provides a semicrystalline glass-ceramic body capable of withstanding high temperatures, which comprises a multiplicity of inorganic crystals dispersed in a glassy matrix comprising the uncrystallised portion of the glass remaining after crystallisation of said crystals, wherein the crystals are formed by crystallisation *in situ*

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from a glass body which is substantially free from TiO_2 and which consists essentially by weight of 35-70% of Al_2O_3, 10-64% of SiO_2, at least one modifying oxide in the indicated proportion selected from 1-20% of P_2O_5, 1-5% of Li_2O, 4-10% of Na_2O, 1-15% of K_2O, 4-25% of Rb_2O, 4-15% of Cs_2O, 4-10% of CaO, 4-10% of SrO, 4-40% of BaO or 4-20% of La_2O_3, the total of the modifying oxides not exceeding 40%, and 0 to 40% of at least one refractory oxide in the indicated proportion of 0-25% of ZrO_2, 0-30% of ThO_2, 0-20% of MgO or 0-10% of BeO.

The glass-ceramic bodies are capable of being used at high temperatures, i.e., at temperatures of 1400°C. and higher and consist essentially of alumina and silica so they are strong, dense, and resistant to thermal shock. The bodies are easy and economical to make using known techniques in an efficient manner.

The addition of a controlled amount the various modifying agents to alumina-silica batches will produce stable glasses containing up to 70 weight percent of alumina which, when given a unique heat treatment, will form a crystalline body containing mullite and/or other high alumina phases and will be useful at temperatures to 1400°C. and higher.

As noted above, binary, high-alumina, aluminosilicate glasses are very unstable. Generally, the phases present, are mullite and beta-cristobalite. Beta-cristobalite is an undesirable component when the alphat-beta cristobalite inversion occurs during the cooling of the body. This inversion commonly causes the crystalline structure to break up, Hence the modifying agents which are added to the glass batch must perform two functions: i.e. they must (1) depress the normal devitrification tendencies of the binary system and produce a relatively stable glass which can be crystallised later by a controlled heat treatment, and (2) inhibit the formation or inversion of cristobalite. We have found compositions in aluminosilicate systems containing phosphorous pentoxide (P_2O_5) and/or the basic metal oxides lithia (Li_2O), soda (Na_2O), potassia (K_2O), rubidia (Rb_2O), cesia (Cs_2O), lime (CaO), strontia (SrO), baria (BaO), and lanthana (La_2O_3), wherein these oxides are present in the amounts given above, (i.e. 1-20% of PaO5, 1-5% of Li_2O, 4-10% of Na_2O, 1-14% of K_2O, 4-25% of Rb_2O, 4-15% of Cs_2O, 4-10% of CaO, 4-10% of SrO, 4-40% of BaO or 4-20% of La_2O_3) and the total amount of such modifiers does not exceed 40% by weight are very satisfactory from the standpoint of glass and glass-ceramic quality and stability.

Throughout this specification, all percentages are by weight. We have further found that a fourth substance, such as one of the refractory oxides zirconia (ZrO_2), thoria (ThO_2), magnesia (MgO), or beryllia (BeO), can, if desired, be added to the glass-forming batch in amounts up to a total of about 40 weight percent and will precipitate a crystal phase compatible with mullite, thereby altering the physical properties as compared to the ternary system. ZrO_2 may be present up to 25% by weight, ThO_2 present up to 30% by weight MgO present up to 20% by weight, and BeO up to 10% by weight. For example, the addition of ZrO_2 to the $BaO{\cdot}Al_2O_3{\cdot}SiO_2$ system results in the precipitation of baddeleyite in association with the mullite and substantially increases the strength of the body and improves its dielectric properties.

In the following Examples, the batches were ball milled to obtain more efficient melting and better glass homogeneity and then melted at 1800-1900°C. in crucibles, pots, or tanks, depending upon the size of melt desired. The melts were poured into moulds, generally fabricated from steel, cooled in air until fairly rigid, introduced into an annealer maintained at 850°C. to 950°C., and then cooled very slowly to room temperature, thus forming bodies of glass. These glass articles were then placed in a furnace and heated at the rate of about 5°C. per minutes up to 1000°-1200°C. and held in that temperature range for 2 hours, in order to initiate crystallisation. It will be understood this rate of heating is purely arbitrary and is chosen to protect the glass shape from thermal shock and deformation. The rate of heating must not be so rapid as to cause breakage of the glass or excessive deformation. The temperature of the furnace was then raised at about 5°C. per minute to 1400°-1600°C. and held in that temperature range for 6-12 hours to obtain the desired crystallisation. The crystallisation of the glass during the reheating cycle proceeds more rapidly as the temperature approaches the liquidus of the crystalline phase. However, in the early stages of crystallisation, the proportion of glassy matrix is large and the article is deformable if its temperature is raised too rapidly. We have found that rate of increase not greater than 5°C./minute gives satisfactory results in most instances. Finally, the crystalline articles were then cooled to room temperature. The rate of cooling to room temperature is dependent upon the body's resistance to thermal shock. Although more rapid cooling has been practiced, we prefer to employ of 5°C./minute cooling cycle. Frequently the heat to the furnace is merely cut off and the furnace allowed to cool at its own rate with the bodies therein.

Where more efficient and economical use of heat is desired the specified heat treatments may be carried out following the shaping of the plastic glass while it is still hot, rather than cooling to room temperature and sub-

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sequently reheating. Thus, after pouring the melt into a mould, or forming the desired shape in any other conventional manner, the body can be cooled rapidly to a temperature below the transformation point, i.e., the temperature at which the liquid glass is considered to become a solid, this temperature being in the vicinity of the annealing point of the glass (for glasses of this invention, a temperature of 850°-950°C) and the required heat treating schedule was then followed.

Furthermore, we have learned that very satisfactory crystalline bodies can be produced where a two-step heat treatment is replaced with a gradual, but constant, increase in temperatures from room temperature or just below the transformation point. This modification in method allows a continuous-type of operation to be carried out. Experience has shown that this rate of heating is dependent upon the thermal character of the glass and the size of the shape involved. Obviously, a rapid rate of heating would be desirable from a commercial standpoint and the low thermal expansion coefficient of our materials has in many cases permitted relatively rapid heating to be pursued. However, as noted above, the rate of temperature increase must be in accord with the rate of crystallisation, otherwise deformation due to a lowering of viscosity will render the final crystalline product generally of little use. Therefore, we prefer a temperature increase of about 5°C/minute to ensure dense crystallisation with little, if any, deformation as the body is heated above the softening point of the glass. Nevertheless, heating rates of 10°C./minute and 20°C/minute and even higher have given useable bodies, particularly if some physical support is given to them. A very slow increase in temperature will also give a satisfactory crystalline product.

Finally, although our preferred practice entails a two-step heating process with a lower heating range of 1000°C-1200°C maintained for 1-4 hours, preferably 2 hours, to initiate crystallisation, followed by a higher heating range of 1400°-1600°C., mainted for 6-12 hours, we have discovered that a usable body can be formed where the glass shape is raised to only just above the transformation point, say 1000°C., and held thereat for a long period of time, e.g. 24 hours and even longer, until the desired crystallisation is attained. If a higher constant temperature is used, the crystallisation rate is more rapid and the crystals are larger. Here again, the rate of crystallisation should preferably substantially balance the rate of deformation of the glass body, i.e., the viscosity of the body cannot be permitted to drop appreciably.

Preferably, a staggered heat treating schedule is followed as total deformation is less where a short holding at a lower temperature is used to begin the heating schedule.

From the foregoing, it can readily be seen that the method of the invention in its simplest terms, involves three steps: (1) melting the batch; (2) cooling the melt below the transformation point; and (3) reheating the glass shape at least to 1000°C., but not more than 1600°C., and holding thereat until the desired crystallisation is attained.

Table I lists examples of bodies produced by our preferred embodiment having compositions of glasses falling within the aforementioned ranges calculated from their respective batches on the oxide basis in weight percent, exclusive of impurities which may be present in the batch materials, as well as listing the melting temperatures. Therefore the batch may be comprised of any materials, either oxides or other compounds, which, on being fused together, are converted to the desired oxide composition in the desired proportions. Table II is a tabulation of the reheating schedule, description of the body, crystal phases present (as determined by X-ray diffraction methods), and some thermal expansion and density measurements.

TABLE I

	1	2	3	4	5	6	7	8
Al_2O_3	40	40	40	44	40	40	40	45
SiO_2	56	50	45	52	55	50	45	40
K_2O	4	10	15	4	—	—	—	—
Rb_2O	—	—	—	—	5	10	15	15
Melting Temp. °C.	1900	1900	1850	1900	1850	1850	1850	1850

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TABLE I (CONTINUED)

	9	10	11	12	13	14	15	16
Al_2O_3	40	40	40	45	40	40	45	45
SiO_2	55	50	45	45	50	45	45	40
Cs_2O	5	10	15	10	—	—	—	—
BaO	—	—	—	—	10	15	10	15
Melting Temp. °C.	1850	1900	1850	1900	1800	1850	1850	1850

	17	18	18	20
Al_2O_3	45	40	45	40
SiO_2	20	20	25	20
K_2O	3	—	—	—
Cs_2O	—	5	—	—
BaO	—	—	5	40
ZrO_2	17	20	15	—
P_2O_5	15	15	10	—
Melting Temp. °C.	1800	1800	1800	1800

	21	22	23	24
Al_2O_3	40	50	40	60
SiO_2	45	35	30	20
K_2O	—	2	—	—
BaO	10	—	—	2
ZrO_2	—	—	20	2
P_2O_5	—	—	10	16
BeO	5	—	—	—
MgO	—	13	—	—
Melting Temp. °C.	1800	1850	1800	1900

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	25	26	27	28	29	30
Al_2O_3	43	45	40	40	40	50
SiO_2	55	50	50	50	45	40
Li_2O	2	—	—	—	—	—
Na_2O	—	5	—	—	—	—
CaO	—	—	10	—	—	—
SrO	—	—	—	10	—	—
La_2O_3	—	—	—	—	15	—
P_2O_5	—	—	—	—	—	10
Melting Temp. °C.	1800	1850	1800	1800	1800	1800

TABLE II

Sample No.	Reheat Schedule	Sample Description	Crystal Phases	Expansion Coeff. × 10^{-7}/°C (25° - 300°C)	Density
1	1120°C - 2 hr. 1520°C - 6 hr.	white, fine grained	millite		
2	1100°C - 2 hr. 1460°C - 8 hr.	white, fine grained	millite	51.9	2.604
3	1110°C - 2 hr. 1500°C - 6 hr.	white, medium to coarse grained	miullte		
4	1120°C - 2 hr. 1520°C - 6 hr.	white, fine grained	mullite		
5	1100°C - 2 hr.	white, fine grained	mullite		
6	1100°C - 2 hr. 1495°C - 6 hr.	white, fine grained	mullite		
7	1100°C - 2 hr. 1495°C - 6 hr.	white, fine grained	mullite		
8	1100°C - 2 hr. 1495°C - 6 hr.	white, fine grained	mullite $Rb_2OAl_2O_34SiO_2$		
9	1100°C - 2 hr. 1510°C - 6 hr.	white, fine grained	mullite		
10	1090°C - 2 hr. 1500°C - 8 hr.	white, fine grained	mullite	33.5	2.824
11	1095°C - 2 hr. 1490°C - 6 hr.	white, fine grained	mullite	40.1	

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TABLE II (CONTINUED)

Sample No.	Reheat Schedule	Sample Description	Crystal Phases	Expansion Coeff. × 10^{-7}/°C (25° - 300°C)	Density
12	1100°C - 2 hr. 1500°C - 6 hr.	white, fine grained	mullite		
13	1100°C - 2 hr. 1535°C - 12 hr.	white, fine grained	mullite	35.6	2.842
14	1090°C - 2 hr. 1500°C - 8 hr.	white, fine grained	mullite	37.8	2.960
15	1090°C - 2 hr. 1500°C - 8 hr.	white, fine grained	mullite	38.5	2.982
16	1110°C - 2 hr. 1500°C - 6 hr.	white, fine grained	mullite $BaOAl_2O_3$		
17	1100°C - 2 hr. 1500°C - 8 hr.	white, fine grained	ZrO_2 mullite	84.5	3.019
18	1100°C - 2 hr. 1500°C - 8 hr.	white, fine grained	ZrO_2 mullite	100.6	3.084
19	1120°C - 2 hr. 1465°C - 6 hr.	white, fine grained	mullite		
20	1100°C - 1-3/4 1500°C - 4-1/2 hr.	white, fine grained	$BaO:Al_2O_3$ $2SiO_2$	69.3	
21	1000°C - 2 hr. 1500°C - 6 hr.	white, fine grained	mullite $BeO:Al_2O_3$		
22	1120°C - 2 hr. 1460°C - 6 hr.	white, fine grained	mullite $MgO-Al_2O_3$		
23	1100°C - 2 hr.	white, fine to medium	mullite ZrO_2		
24	1120°C - 2 hr. 1515°C - 6 hr.	white, fine grained	mullite		
25	1100°C - 2 hr. 1450°C - 6 hr.	white, fine grained	mullite	37.2	2.656
26	1095°C - 2 hr. 1490°C - 6 hr.	white, fine grained	mullite	45.9	
27	1100°C - 2 hr. 1450°C - 6 hr.	white, fine grained	mullite		
28	1100°C - 2 hr. 1535°C - 12 hr.	white, fine grained	mullite	38.5	
29	1100°C - 2 hr. 1500°C - 6 hr.	white, fine grained	mullite		
30	1100°C - 2 hr. 1450°C - 6 hr.	white, fine grained	mullite		

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The above tables demonstrate that crystalline bodies containing mullite and/or other high alumina phases can be obtained from high alumina, aluminosilicate glasses where a minor amount of a modifying agent is added. The average modulus of rupture was greater than 10,000 psi for the crystalline bodies.

The quantities of alumina and modifier present are critical to the invention as at least 35 weight percent of Al_2O_3 must be present to ensure a substantial crystallisation of mullite and/or other high alumina materials, but a stable glass cannot be formed where more than 70 weight percent of Al_2O_3 is present. At least the minimum amount stated of the modifying agents must be available to produce a stable glass and inhibit the formation of cristobalite, but if more than the designated amount of each (or most than total of 40 percent of a combination of them) is introduced, crystals are formed which are incompatible with the mullite and other high alumina phases. This incompatibility may involve a great difference in the coefficients of thermal expansion between the high alumina crystallisation and that of the extraneous phase introduced with an excess of a modifying agent, such that the body will be disrupted during the heat treatment. This incompatibility may also involve the formation of large crystals of the extraneous phase during heat treatment such that the desired fine-grained structure which ensures high strength and density in the final product, is not possible.

Likewise, the amounts of the refractory oxides which can be tolerated must be strictly observed to ensure the production of a stable glass followed by the formation of crystals compatible with the high alumina phases upon heat treatment. Finally we have found that while a maximum of as much as 64 weight percent of SiO_2 may be present, at least 10 weight percent must be present to ensure glass stability.

As has been explained above, no separate nucleating agent as such is required for this invention. The crystals formed are small, substantially all being finer than 30 microns, and randomly oriented. Laboratory test have indicated the crystal content of the body to be at least 25% by weight and generally over 30% by weight, this depending upon the extent to which components of the batch are adaptable to the formation of crystalline phases. Moreover, as can be seen from Tables I and II the invention provides dense, strong bodies which are resistant to thermal shock and which are useable at temperatures over 1400°C. As the final crystalline body is formed from a non-porous glass it is non-porous, thereby greatly increasing its resistance to molten substances or gases with which it may be brought into contact.

In the accompanying drawings Figs. 1-10 illustrate the ranges of the fundamental three-component compositions encompassed in our invention wherein Fig. 1 includes P_2O_5 as the modifying agent; Fig. 2 includes Li_2O as the modifying agent; Fig. 3 includes Na_2O as the modifying agent; Fig. 4 includes K_2O as the modifying agent; Fig. 5 includes Rb_2O as the modifying agent; Fig. 6 includes Cs_2O as the modifying agent; Fig. 7 includes CaO as the modifying agent; Fig. 8 includes SrO as the modifying agent; Fig. 9 includes BaO as the modifying agent; and Fig. 10 includes La_2O_3 as the modifying agent.

Fig. 11 records a time-temperature curve for the heat treatment of a specific example of the invention, namely Example 12 given in Table II wherein, after the glass-forming batch had been melted, shaped, and cooled to room temperature, the shape was given the following heat treatment: the temperature was raised at 5°C./minute to 1100°C., maintained thereat for 2 hours, thereafter the temperature was raised at 5°C./minute to 1500°C., held thereat for 6 hours, and then the body was cooled at 5°C./minute to room temperature. The area enclosed within the dotted lines represents the ranges of time and temperature of our preferred two-step process.

WHAT WE CLAIM IS:

1. A semicrystalline glass-ceramic body capable of withstanding high temperatures, which comprises a multiplicity of inorganic crystals dispersed in a glassy matrix comprising the uncrystallised portion of the glass remaining after crystallisation of said crystals, wherein the crystals are predominantly of high alumina compounds and are formed by crystallisation *in situ* from a glass body which is substantially free from TiO_2 and which consists essentially by weight of 35-70% of Al_2O_3, 10-64% of SiO_2, at least one modifying oxide in the indicated proportion selected from 1-20% of P_2O_5, 1-5% of Li_2O, 4-10% of Na_2O, 1-15% of K_2O, 4-25% of Rb_2O, 4-15% of Cs_2O, 4-10% of CaO, 4-10% of SrO, 4-40% of BaO, or 4-20% of La_2O_3, the total of the modifying oxides not exceeding 40% and 0 to 40% of at least one refractory oxide in the indicated proportion of 0-25% of ZrO_2, 0-30% of ThO_2, 0-20% of MgO of 0-10% of BeO.

2. A semicrystalline glass-ceramic body as claimed in claim 1, wherein substantially all of the crystals are smaller than 30 microns in diameter and comprise at least 25% by weight of said semi-crystalline body.

3. A method of manufacturing a semicrystalline glass-ceramic body capable of withstanding high temperatures which comprises the steps of melting a glass-forming composition which is substantially free from

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TiO_2 and which consists essentially by weight of 35-70% of Al_2O_3, 10-64% of SiO_2, at least one modifying oxide in the indicated proportion of 1-20% of P_2O_5, 1-5% of Li_2O, 4-10% of Na_2O, 1-15% of K_2O, 4-25% of Rb_2O, 4-15% of Cs_2O, 4-10% of CaO, 4-10% of SrO, 4-40% of BaO or 4-20% of La_2O_3, the total of said modifying oxides not exceeding 40% and 0 to 40% of at least one refractory oxide in the indicated proportion of 0-25% of ZrO_2, 0-30% of ThO_2, 0-20% of MgO and 0-10% of BeO, simultaneously cooling the melt below the transformation point of the melt and forming a glass shape therefrom, exposing the shape to a temperature of at least 1000°C, but not more than 1600°C, maintaining thereat for a time sufficient to attain the desired crystallisation, and thereafter cooling the shape to room temperature.

4. A method as claimed in claim 3, wherein, the exposing of the shape to a temperature of at least 1008°C but not more than 1600°C is carried out by increasing the temperature of said shape at a rate whereby the rate of crystallisation substantially balances the rate of deformation of the shape.

5. A method as claimed in claim 4, wherein the rate of heating is about 5°C/minute.

6. A method as claimed in any of claims 3 to 5, wherein the shape is exposed to a temperature of 1000°C. to 1200°C for 1 to 4 hours and then the temperature is raised to 1400°C. to 1600°C and maintained thereat for 6 to 12 hours.

7. A method as claimed in claim 3, substantially as described with reference to any of the Examples.

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Fig. 1

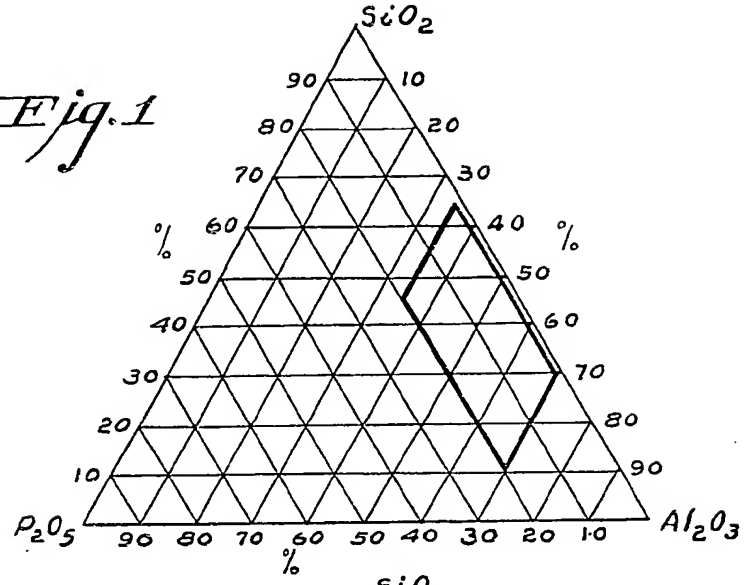

Fig. 2

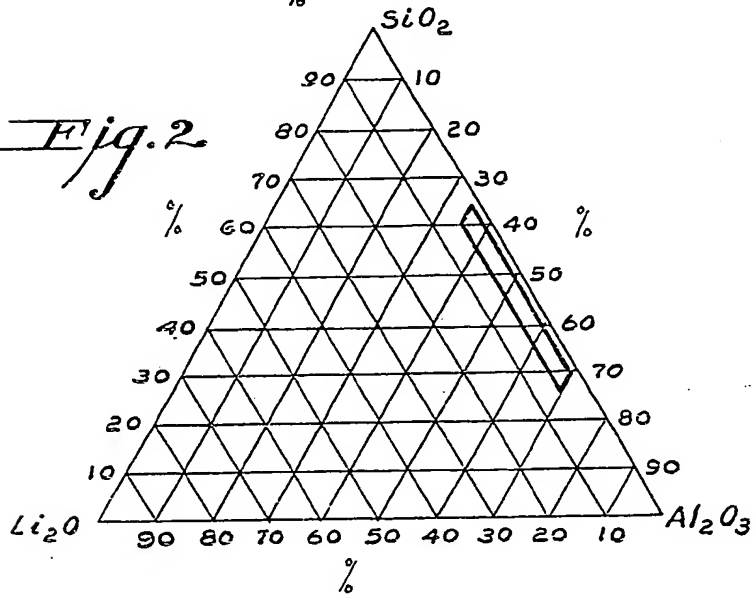

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Fig. 3

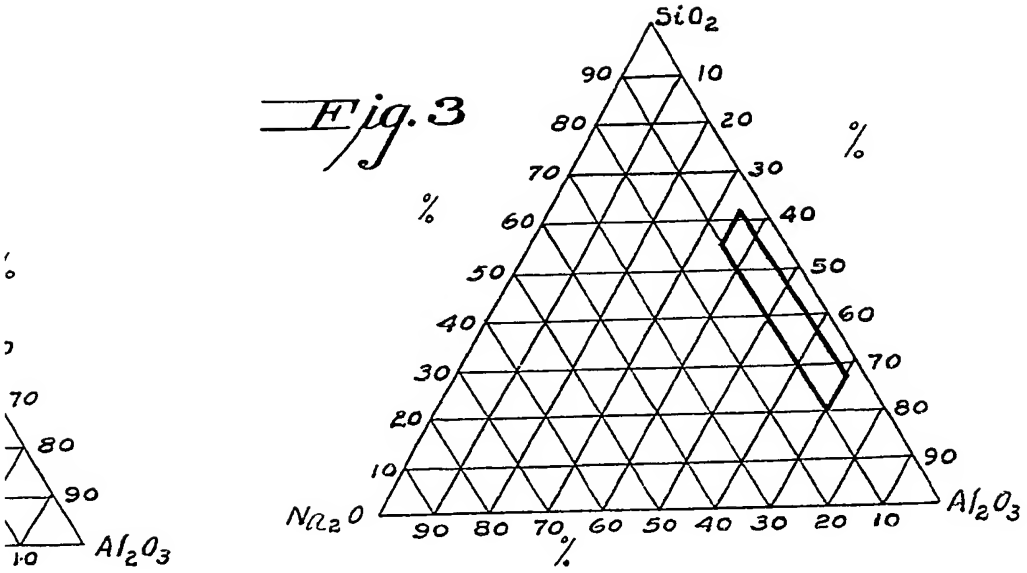
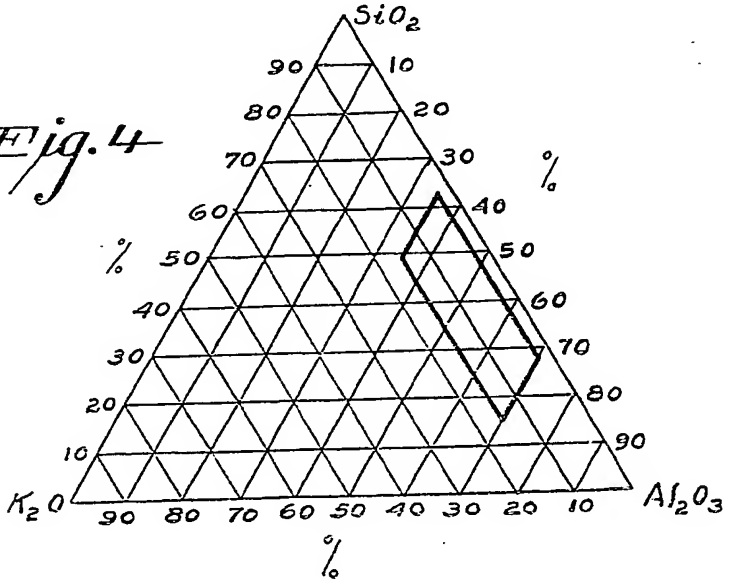


Fig. 4



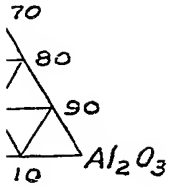


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Fig. 1

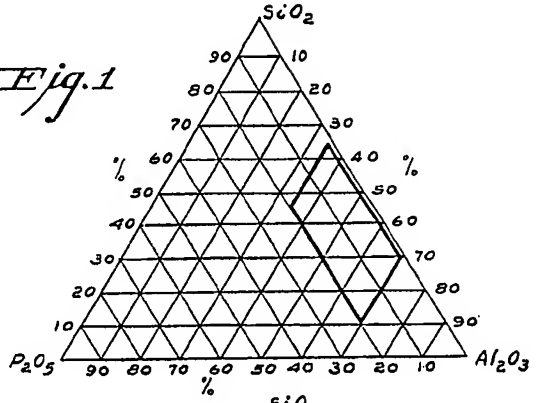


Fig. 2

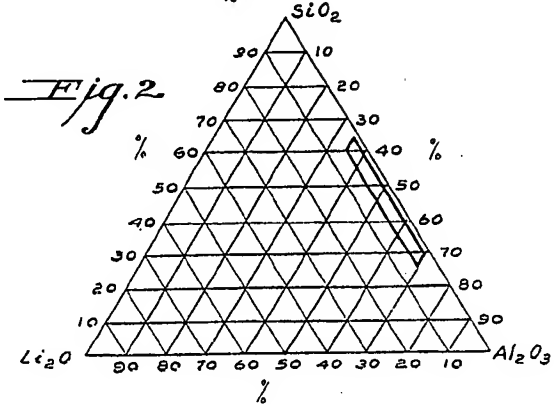
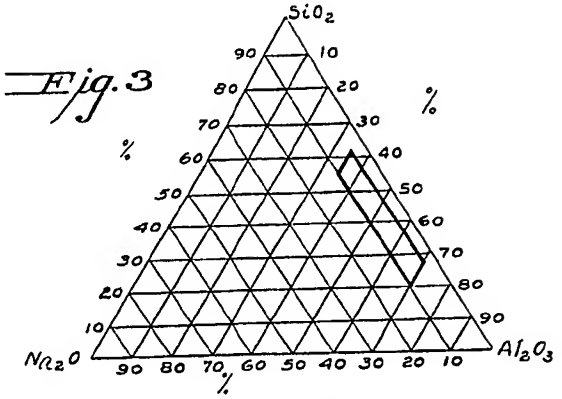


Fig. 3



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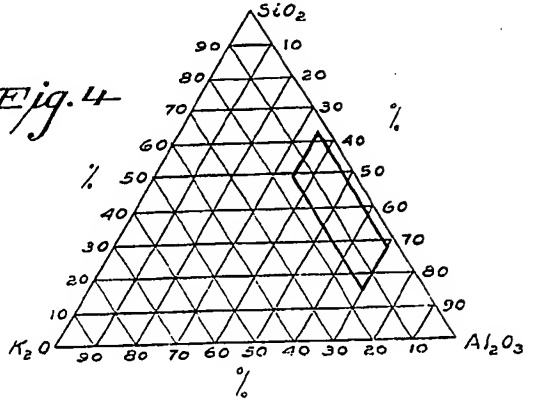

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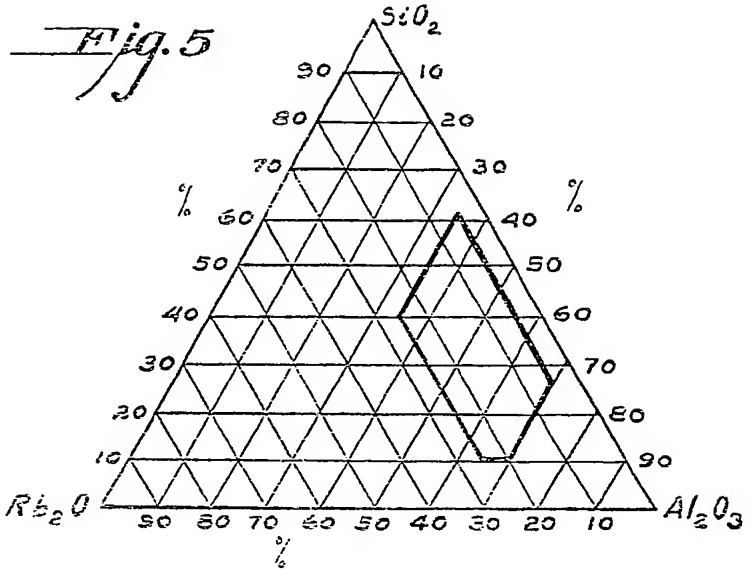

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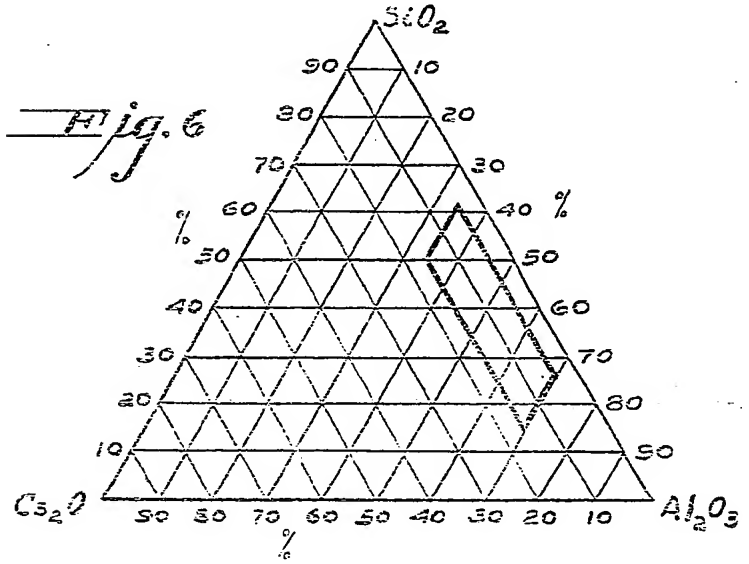

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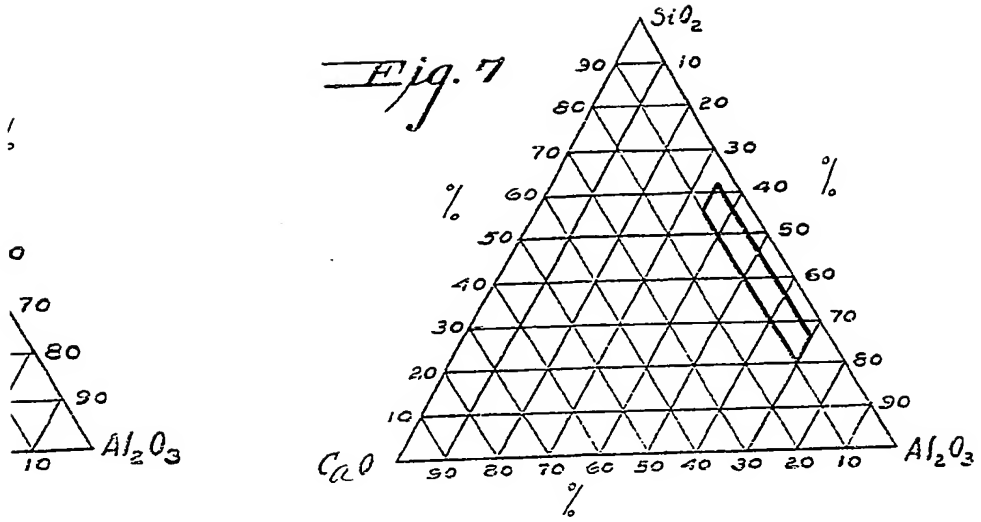
1005338 COMPLETE SPECIFICATION
6 SHEETS This drawing is a reproduction of the Original on a reduced scale
Sheets 3 & 4

Fig. 7



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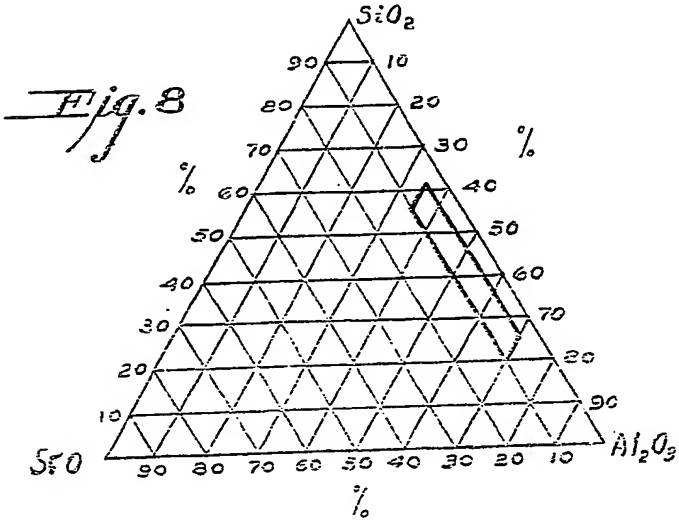

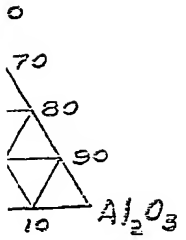

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1005338 COMPLETE SPECIFICATION
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Sheets 3 & 4

Fig. 5

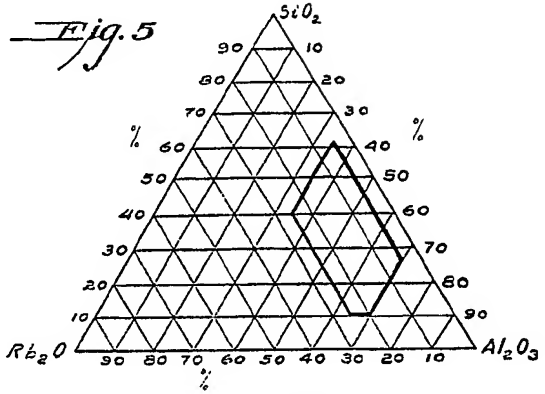
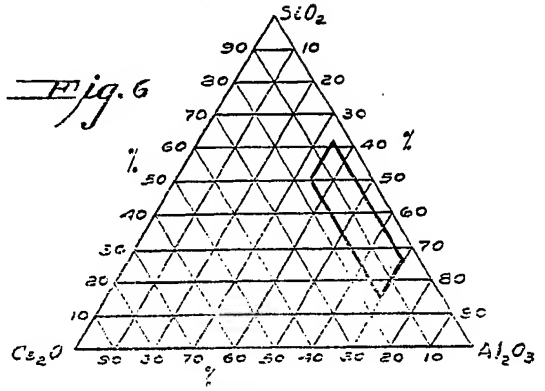


Fig. 6



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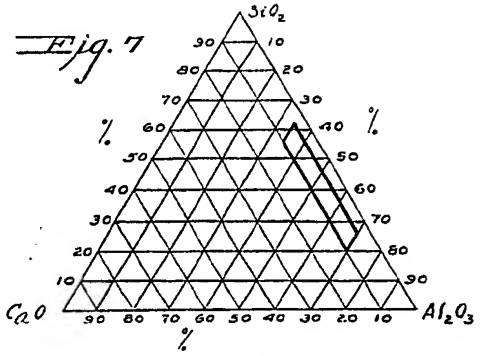

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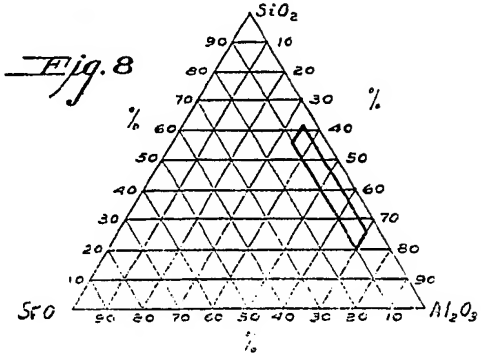


Fig. 9

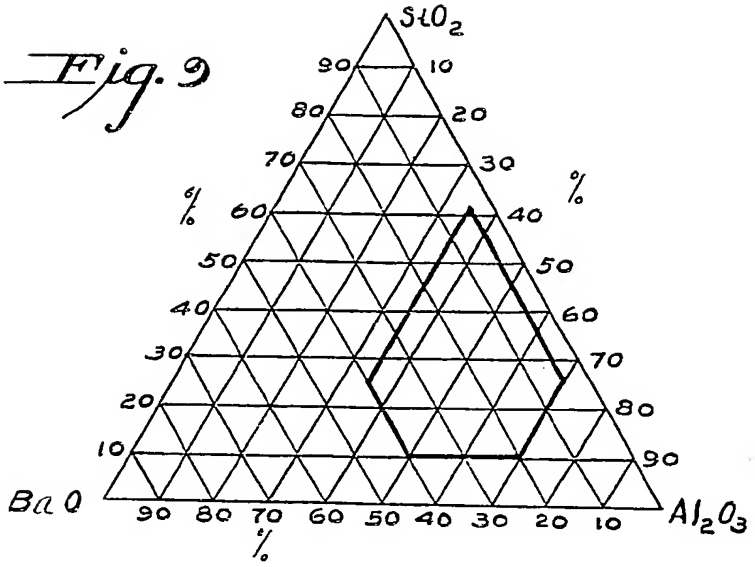
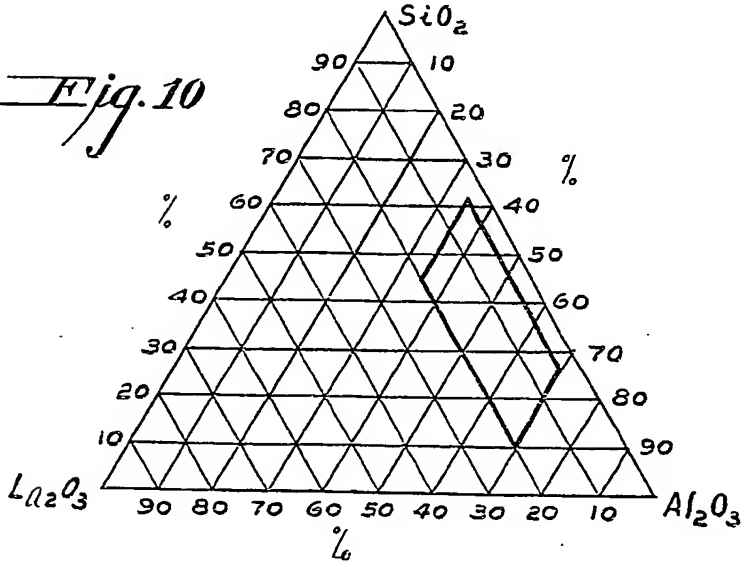


Fig. 10



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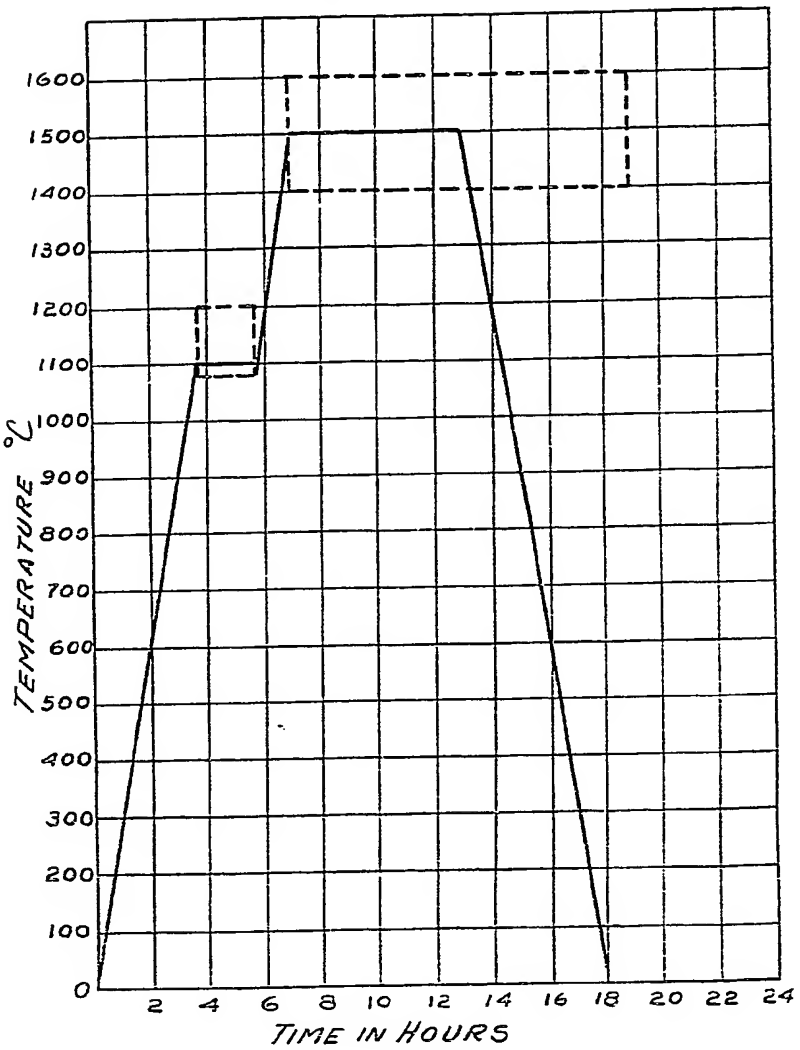
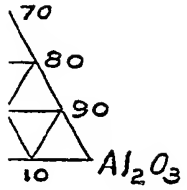
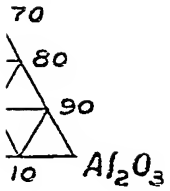
1005338 COMPLETE SPECIFICATION
6 SHEETS *This drawing is a reproduction of the Original on a reduced scale*
Sheets 5 & 6

Fig. 11







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1005338 COMPLETE SPECIFICATION
6 SHEETS This drawing is a reproduction of the Original on a reduced scale
Sheets 5 & 6

Fig. 9

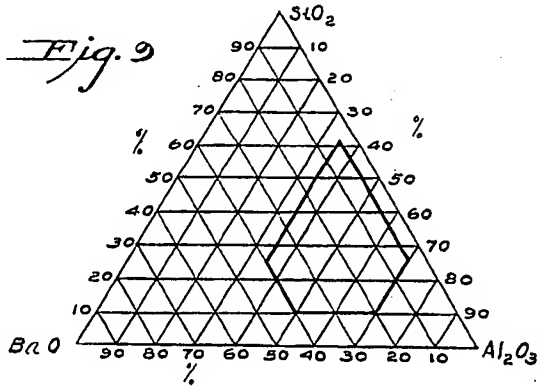


Fig. 10

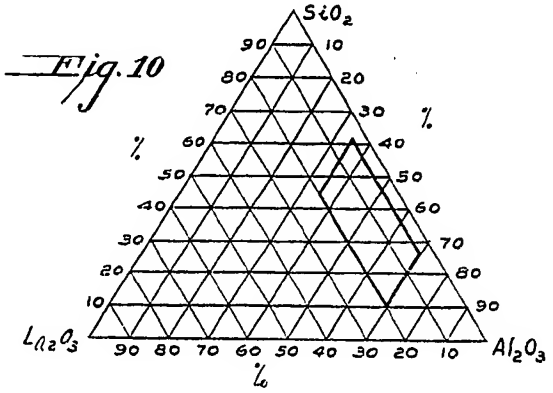
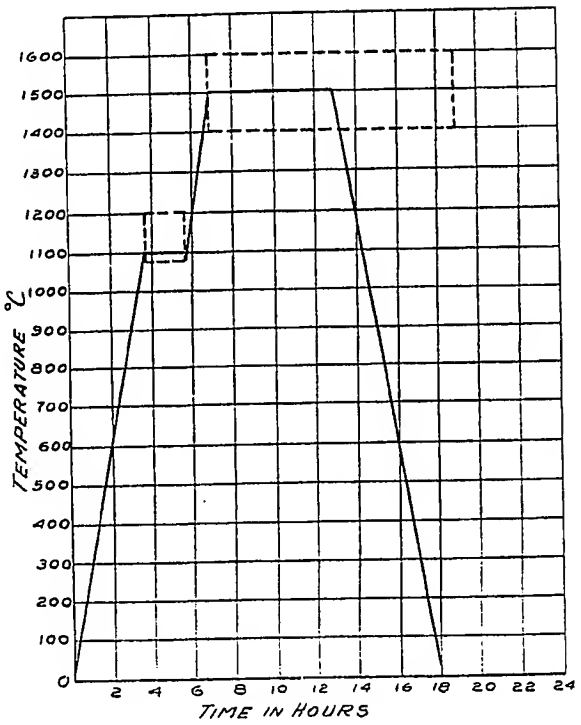


Fig. 11



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